CHAPTER 8

INSTRUMENT SETUP AND SAMPLE ANALYSIS - RESONANCE MARAN ULTRA PULSED WAVE NMR ANALYZER

8.1 INSTRUMENT SETUP

a. Setup Information.

Operators must read the user's manual and familiarize themselves with this instruction before operating the NMR instrument. Any error messages should be referenced in the Operator's manual.

The operation of the Resonance MARAN Ultra instrument is controlled by a personal computer. After the instrument is powered on, a series of automatic internal tests are carried out to confirm the instrument communications and the functionality of the console unit.

The internal magnet is maintained at a temperature of 40 Degrees Centigrade. The unit must not be used for a period of 12 hours after being switched on to allow the magnet temperature to stabilize. After being switched-on the instrument should remain on.

(1) Open the desktop folder "EasyCal Applications."

(2) Start the "EasyCal for Oilseeds" software by double-clicking on the icon.

NOTE: Alternatively, select the icon with a left mouse click, followed by a right mouse click and selecting the "Open" option in the pop-up menu.

(3) Enter the administrative password to get access to the calibration software and click "OK" to enter the software. "MARAN" is the password installed at the factory.

(4) Insert the tuning sample (100 grams of vegetable oil) to begin the parameter optimization and verification of instrument performance. The instrument will optimize the instrument parameters in the order of resonance frequency, pulse width, and instrument sensitivity. Once the software has completed one cycle of these tests it will perform the same tests a second time.

(5) When the second cycle of tests is completed, record the operating parameters: Frequency Offset (Auto Tuning or 01), RF Pulse Width (Autoset P90), and Receiver Gain (Autoset RG) on the calibration log (Attachment A).

If the difference between the new values for P90 and RG and the previous results are greater than 5 percent of the previous reading, the user should stop the setup process and contact Resonance for technical support.

NOTE: Clicking on the "Results" button will display a comparison of the current and previous parameters.

(6) Click "Continue" to begin the calibration procedure.

b. Calibration.

(1) "GIPSA Dry Seed" for the name of the calibration and click "OK".

(2) Enter the sample ID, weight, and oil content of the High Sunflower Seed Standard (SSS) and click the "Continue" button.

(3) Insert the High SSS.

NOTE: The instrument sensitivity will be optimized base on the sample highest in oil content. During the first part of the measurement, "EasyCal" maximizes the receiver gain (RG) parameter (adjustable from 0 - 100%) to a maximum value. Once the receiver sensitivity is optimized, the NMR signal is recorded for the high standard.

(4) When the measurement is complete, remove the High SSS.

(5) Click "Yes" to measure the second standard.

(6) Enter the sample ID, weight, and oil content of the Low SSS and click the "Continue" button.

(7) Insert the Low SSS.

(8) Once the calibration standard has been measured, click "No" and remove the Low SSS from the magnet.

(9) Select the "Automatic" calibration method option. The two measured points are plotted as NMR signal/gram of seeds versus oil content, along with the statistics of the calibration. Record the value intercept, concentration intercept, and slope on the calibration log.

(10) Click "Yes" to accept the calibration.

NOTE: If prompted to confirm overwriting of the existing calibration, click yes.

(11) Click on the "Exit" button to leave the calibration program.

(12) Click "Yes" to confirm that you wish to terminate the "EasyCal Calibration" program.

c. Calibration Check.

Check the calibration by testing the SSS as market samples before beginning routine sample analysis.

(1) Open the desktop folder titled "EasyCal Calibrations" by double clicking on it.

NOTE: All calibrations generated using the"EasyCal" software will be found in this folder.

(2) Double click on the "GIPSA Dry Seed" calibration icon.

NOTE: Before measuring seeds, the instrument will perform tuning and diagnostics to verify the instrument is performing within specifications.

(3) Insert the tuning sample (100 grams of vegetable oil sample) in the instrument to begin the automatic tuning procedure.

NOTE: The tuning procedure consists of first checking the instrument frequency. Once the 4 scan sequence is completed, the software will display a window of the old value, new value, and the difference. The software will then automatically proceed to measure the signal intensity for the tuning sample. The software will record the signal intensity in a diagnostic file. These values are recorded in a running file MQDiagnosis.log in the following directory

C:\Program\Files\Resonance\RlCalib\bin

(4) Record the new tuning frequency value (01) on the check sample log and click "Yes" to accept the new value.

(5) Remove the tuning sample and click on the "Start Analysis" button.

(6) Enter the ID and the weight of the High Sunflower Seed Standard (SSS) and click "OK".

(7) When prompted, insert the High SSS.

(8) When the analysis is complete, remove the SSS, record the oil value on the check sample log, and calculate the difference between the reported value for the SSS and the known value. If the difference is greater than 0.3, repeat the analysis. If the repeat analysis result difference is still greater than 0.3, recalibrate the instrument.

(9) Repeat steps 6 - 8 with the Low SSS.

NOTE: Test the SSS as a market sample to check the NMR instrument accuracy after calibration, when the room temperature changes by ± 0.5° C, after every 30 - 40 samples have been analyzed, or every two hours, whichever comes first. Maintain a record (electronic or written) of the calibration checks using the Check Sample Log in Appendix B as a template.

(10) After completing the check sample procedure, follow the procedures listed below to begin analyzing market samples.

8.2 SAMPLE ANALYSIS

a. Analyzing Samples.

For each new sample, a window for entering the sample details will be presented. Follow the steps listed below for analyzing a sample.

(1) Enter the sample mass.

NOTE: The sample mass can be entered manually, or if an electronic scale is connected, via the weigh button.

(2) Enter the Sample ID and click "OK" to proceed.

(3) Insert the sample in the instrument. The instrument will display the oil content when the analysis is completed.

NOTE: The sample ID, oil content, time and date of analysis will be logged to a log file. All log files are located in the following subdirectory:

C:\Program Files\Resonance\RlCalib\bin

(4) Repeat steps (1) - (3) for each sample analyzed. Once the last sample is analyzed and removed from the analyzer, click the "CANCEL" button and then select "QUIT".

(5) Click "Yes" to terminate the "Rl Analysis" Program.

b. Reporting Results.

Record and report the percent oil on the pan ticket, inspection log, and certify to the nearest tenth percent using the standard FGIS rounding procedures.